

Osmotic Dehydration as a Tool for Industrialization of Jabuticaba Peel (*Myrciaria jabuticaba*)

Lismaira Goncalves Caixeta Garcia¹, Franciello Vendruscolo², Flavio Alves da Silva³, Ellen Caroline Silverio Vieira², Thays Lorrayne Lavrinha e Silva², Clarissa Damiani²

¹Agronomy Departament, Federal University of Goiás, Goiânia, Goiás, Brazil.

²Food Technology Departament, Federal University of Goiás, Goiânia, Goiás, Brazil.

³Agronomy Departament and Food Science Departament, Federal University of Goiás, Goiânia, Goiás, Brazil.

Abstract— This study evaluated the osmotic dehydration of jabuticaba peel for use as a by-product, with development of new food products. Response surface methodology was used, considering temperature and sucrose concentration as independent variables, assessing their effects on water loss, solid gain, mass loss, and solid gain rate. Sucrose concentration had a greater influence on osmotic process. Temperature increase is necessary in osmotic dehydration, once it leads to tissue softening, which is essential for dehydration of jabuticaba peel. Therefore, the best osmotic dehydration conditions were set at 60°C and 70 °Brix. With respect to the physicochemical characterization of the bioactive compounds of dehydrated jabuticaba peel, considerable amounts of sugars, anthocyanins, and phenolic compounds were observed, besides the antioxidant potential. Thus, dehydration of jabuticaba peel is a viable alternative to minimize the waste generated during harvest, being a product with high nutritional value.

Keywords— antioxidant potential, by-product, phenolic compounds, processing.

I. INTRODUCTION

Jabuticaba tree (*Myrciaria* sp) belonging to Myrtaceae family is among the most important native species in Brazil. Although it is a plant originating from Minas Gerais, it grows in almost all Brazilian regions, and in other countries such as Bolivia, Argentina, Uruguay, and Peru [1,2]. It is intensely cultivated, and very productive, having small fruits with thin flesh, black epicarp, very tasty, and characterized by early maturation [3]. Despite its popularity in Brazil, jabuticaba does not have high commercial value, once it is a very perishable fruit, with limited period for consumption after harvesting. The fruit has a shelf life of up to three days, when changes are observed in appearance, due to the intense water loss and pulp fermentation, which hinders its commercialization [4]. To prevent losses, the fruit can be industrialized,

which generates appreciable amounts of peel and seeds, representing approximately 50% of the fruit.

Osmotic dehydration is among the techniques employed for postharvest conservation of agricultural products, also called dehydration by immersion, consisting basically of water removal from the food by the effect of osmotic pressure, which occurs by immersion the product in hypertonic solution containing one or more solutes, at predetermined time and temperature. It is used as alternative for the production of dehydrated fruits, providing suitable texture, color, and flavor, besides reducing postharvest losses [5,6], transportation costs, with easier packing and storage [7].

The development of new products using by-products from the food industry, such as peels, is a tendency, not only for their rich nutritional value, but also to prevent the accumulation of residues. Thus, the objective of this study was to evaluate the osmotic dehydration of jabuticaba peel, in order to make better use of the by-product, and develop new food products.

II. MATERIAL AND METHODS

2.1. Raw material

Jabuticaba fruits, from *Myrciaria jabuticaba* (Vell) Berg species, crop 2012, were harvested at the farm Fazenda e Vinícola Jabuticabal, in Nova Fátima, district of Hidrolândia, located at 16° 55' 32.35" South latitude and 49° 21' 39.76" West longitude, in the State of Goiás, Brazil. Fruits were selected, washed with clean water, sanitized with sodium hypochlorite at 100 µL L⁻¹ for 15 minutes. Peels were obtained by electrical depulping machine (Itametal, Bonina 0.25 df), and pulp and seeds were discarded.

2.2. Osmotic dehydration

The osmotic dehydration of jabuticaba peels was performed at different temperatures and sucrose concentrations, in thermostatic bath (Marconi, Brazil), with 80 ± 5 revolutions per minute, with a 1:4 (w/w)

peel:osmotic solution ratio. The experiments lasted 6 hours, and moisture and total soluble solids contents were determined every hour, according to AOAC [8], and the results were used to calculate water loss, solids gain, mass loss, and solid gain rate, according to the Equations 1 to 4, respectively:

$$WL=100. \left[1 - \left(\frac{M_f \cdot X_i}{M_i \cdot X_f} \right) \right] \quad (1)$$

where, WL is water loss in relation to the initial mass (%); M_i is the initial mass (g); M_f is the final mass (g); X_i is the initial moisture on a wet basis (%), and X_f is the final moisture on a wet basis (%).

$$SG=100. \left[\frac{(SS_f \cdot M_i) - (SS_i \cdot M_f)}{M_i} \right] \quad (2)$$

where, SG is total soluble solids gain in relation to the initial mass (%) SS_i is the initial total soluble solids (°Brix), and SS_f is the final total soluble solids (°Brix).

$$ML=WL-SG \quad (3)$$

where, ML is the mass loss relative to the initial mass (%).

$$SGR= \frac{SS_2 - SS_0}{2} \quad (4)$$

where, SGR is the solid gain rate in the first 2 hours of osmotic dehydration (°Brix h⁻¹), and SS_2 is the sucrose concentration after 2 hours of osmotic dehydration (°Brix).

2.3. Experimental Design and Statistical Analysis

The effects of temperature and sucrose concentration on WL, SG, ML, and SGR responses were analyzed by response surface methodology (Table 1). All data were adjusted to a second-order mathematical model (5), to correlate them with independent variables.

$$y=\beta_1+\beta_2 T+\beta_3 T^2+\beta_4 C+\beta_5 C^2+\beta_6 TC \quad (5)$$

where, y is the response of each dependent variable (WL, SG, ML, SGR); β_1 is a constant; β_2 is the linear coefficient of temperature; β_3 is the quadratic coefficient of temperature; β_4 is the linear coefficient of sucrose concentration; β_5 is the quadratic coefficient of sucrose concentration ; β_6 is the interaction coefficient of temperature and sucrose concentration, T is the temperature, and C is the sucrose concentration. The linear, quadratic, and interaction effects of temperature and sucrose concentration on the responses, as well as the experimental error, t coefficient, and statistical significance p were estimated by the software Statistica 7.0. Analysis of variance (ANOVA) was used to analyze statistical significant differences under the experimental conditions.

2.4. Drying process

Drying of jabuticaba peel was performed in a convective dryer (1.90 m high and 0.80 m wide, with five metal trays of 0.055 m 0.057 m), at 60 °C and air flow rate of 0.0206 m³ kg⁻¹ s⁻¹, until the product reached a final moisture of 20 to 25%, and water activity between 0.5 and 0.6.

2.5. Physicochemical characterization

The moisture content was determined by oven drying at 105 °C until constant weight; ash content was measured by gravimetric method after incineration in a muffle furnace at 550 °C; total nitrogen was determined by Kjeldahl method, considering the conversion factor of 6.25 for crude protein, according to AOAC [8]. Total lipids were determined by the method of Bligh and Dyer [9], based on the mixture of three solvents: water, methanol and chloroform. Total carbohydrates were calculated by subtracting the protein, lipids, ash, and moisture from 100. Reducing sugars were determined by the 3,5-dinitrosalicylic method [10]. TSS content was performed by °Brix readings of the sample at 20 °C in a digital refractometer (Atago N-1E); pH was measured with a digital potentiometer (pH Meter HI-9224); total acidity was assessed by titration with 0.1 N NaOH [8]. Water activity was measured using an Aqualab (Aqualab CX-2) apparatus at 25 °C. Color determination was performed in colorimeter (Hunterlab, ColorQuest II) by measuring the coordinates L*, a*, and b*.

2.6. Bioactive compounds

The antioxidant activity was measured by DPPH (2,2-diphenyl-1-picrylhydrazyl) assay [11], with modifications by Borguini and Torres [12]. The degree of discoloration of the DPPH radical was measured in spectrophotometer at 517 nm after 20 minutes of reaction (Biospectro SP-220).

Phenolic compounds, expressed as mg of gallic acid equivalents (GAE), were determined in spectrophotometer (Biospectro SP-220) at 750 nm, using the Folin-Ciocalteu reagent [13]. The antioxidant activity and phenolic compounds were determined in the extracts using three solvents of different polarities, ether (2.9), ethanol (5.2), and water (9).

The total anthocyanins were determined in spectrophotometer (Biospectro SP-220), at 535 nm by the method of Lees and Francis [14], adjusted by Barcia et al. [15]. The quantification of anthocyanins was based on molar absorption coefficient of cyanidin-3-glucoside (6), which is the major anthocyanin present in fruits,

$$Abs=\epsilon \cdot C \cdot l \quad (6)$$

where, Abs is the absorbance; ϵ is the molar absorption coefficient (L mol⁻¹ cm⁻¹); C is the concentration (mol L⁻¹), and l is the optical path length (cm).

III. RESULTS AND DISCUSSION

3.1. Osmotic Dehydration

Sucrose concentration significantly affected the parameters WL, SG, and SGR during the osmotic dehydration of jabuticaba peel, when compared to temperature (Table 2). Higher mass losses were observed when using solutions at 67 to 75 °Brix. Besides the search for incorporating solids in jabuticaba peel, reducing water loss due to formation of a sucrose barrier, water loss plays

a role in food preservation, by reducing the moisture content and water activity of the product, therefore requiring less time for the application of the secondary preservation method.

Increases in both sucrose concentration and temperature resulted in higher SGR, obtaining results similar to those at longer times. On the other hand, lower SGR promotes resistance to water loss. However, temperature can play an indirect effect on osmotic dehydration, since above 60 °C, structural characteristics are modified by increasing the cell membrane permeability, allowing the impregnation of solids [16]. Aktas et al. [17] studied the osmotic dehydration of apples, and found that an increase in sucrose concentration of the osmotic solution led to a decrease in dehydration time. Thus, temperatures above 60 °C should be used for dehydration of jabuticaba peel, aiming both impregnation of solids and tissue softening.

A significant linear effect of temperature (Table 3) ($p \leq 0.05$) was observed only for SGR, and a significant quadratic effect ($p \leq 0.05$) for WL. The most significant linear effect ($p \leq 0.05$) was observed for the variable sucrose concentration for the responses WL, SG, and SGR, with quadratic effect ($p \leq 0.05$) for WL and ML. Only the significant p-values were considered, and ANOVA (Table 4) was used to evaluate the significance and the lack-of-fit of the second-order polynomial regression (Eq. 5) by F-test.

Adjusted polynomial models of WL, SG, ML, and SGR presented significant regression ($p \leq 0.05$), with calculated F higher than tabulated F, as shown in Table 5, and the response surface of WL, SG, ML and SGR as a function of the independent variables are presented in Fig 1. High coefficients of determination (R^2) were obtained for WL and SGR, with values of 0.7525 and 0.7181, respectively.

As reported by several authors, the increase in sucrose concentration can lead to water loss due to the concentration gradient. Duarte et al. [18] studied dehydration of jackfruit slices using sucrose solutions (40 to 50 °Brix), and observed higher water loss when using osmotic solution at 50 °Brix. In contrast, Mercali et al. [19] investigated the osmotic dehydration of blueberries, and found that water loss was favored by higher temperatures rather than sugar concentrations.

3.2. Physicochemical characterization, energy value, and bioactive compounds

The proximate composition of dehydrated jabuticaba peel is shown in Table 6. The jabuticaba peel of the present study was within the acceptable standards of the Brazilian law, which has established maximum 25% moisture content in dehydrated fruits.

The reducing and non-reducing sugars, total soluble solids, pH, titratable acidity, and water activity of dehydrated jabuticaba peel are shown in Table 6. Despite

the use of sucrose in the osmotic dehydration, sugar levels were higher than the non-reducing sugars, probably due to the dissolution of sucrose in water during the process. According to Bobbio and Bobbio [20], sucrose in acidic aqueous medium, such as the osmotic medium (pH 3.41) undergoes hydrolysis to the reducing monosaccharides D-glucose and D-fructose. Furthermore, the pH values below 4.5 of this study ensure food safety without the need for very high temperature treatments.

With respect to the color parameters, the L^* , a^* , and b^* values of jabuticaba peel were 27.7467 ± 0.1882 , 1.0667 ± 0.0493 , and -0.4633 ± 0.0208 , respectively. The final product was black colored, as expected in maturity stage.

The anthocyanin content of the dehydrated jabuticaba peel was lower (22.0893 ± 0.1402 mg cyanidin-3-glucoside 100 g^{-1}) than that found by Misugi and Rosso [21] in jabuticaba peel *in natura*, who found $59.62 \text{ mg } 100 \text{ g}^{-1}$.

The extraction process using solvents with different polarities allowed the extraction of phenolic compounds in varying amounts. The aqueous extract exhibited higher phenolic content (Table 6), when compared to ether and ethanolic extracts. This difference suggests the effect of the solvent on the phytochemicals profile of the sample. By presenting different degrees of polymerization, phenolics are extracted according to their solubility in pure or diluted organic solvent [22]. According to Pellegrini et al. [23] and Melo et al. [24], the solubility in a given solvent is an intrinsic characteristic of a given phytochemical, which explains the absence of a universal extraction procedure due to the structural diversity and sensitivity of phenolic compounds to extraction conditions. The aqueous extract showed higher levels of phenolic compounds, thus higher antioxidant potential, when compared with the other solvents (ether and ethanol), probably due to the protective effect of antioxidants is highly related to the presence of phenols and anthocyanins in fruits and vegetables.

IV. CONCLUSION

Osmotic dehydration of jabuticaba peel is a viable alternative to minimize the waste generated during harvest. In addition, it is a product with high nutritional value, showing considerable amounts of anthocyanins, phenolic compounds, and antioxidants. During osmotic dehydration of jabuticaba peel, the concentration of the osmotic solution provided mass transfer between the fruit and the solution, resulting in higher water loss, solids gain, mass loss, and solids transfer rates. However, temperatures around 60 °C should be used for providing tissue softening and solids incorporation. Therefore, the best condition for osmotic dehydration of jabuticaba peel was osmotic solution concentration of 70 °Brix and temperature of 60 °C.

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Table.1: Real and encoded variables studied in Response Surface Methodology (RSM).

Variable	Levels of variation ¹				
	-1.414	-1	0	+1	+1.414
Temperature (°C)	35.86	40	50	60	64.14
Sucrose concentration (°Brix)	45.86	50	60	70	74.14

¹±1.414 corresponds to $\pm\alpha$ ($\alpha=\sqrt{2}$).

Table 2: Mean values and standard deviation of WL, SG, ML and SGR obtained by the RSM of osmotic dehydration of jabuticaba peel.

Temperature (°C)	Sucrose concentration (°Brix)	WL (%)	SG (%)	ML (%)	SGR (°Brix h ⁻¹)
40	50	47.042±0.801	12.850±0.292	34.192±0.524	0.100±0.000
40	70	54.861±2.532	17.284±1.502	37.548±3.799	0.133±0.003
60	50	48.232±1.864	8.875±0.4988	39.357±2.349	0.100±0.000
60	70	57.836±3.623	12.456±0.695	45.369±4.263	0.160±0.000
35.86	60	59.769±2.486	8.678±0.192	51.092±2.312	0.100±0.000
64.14	60	62.143±0.254	16.866±1.420	45.369±1.348	0.140±0.000
50	45.86	48.018±1.400	5.813±1.646	42.205±2.065	0.102±0.003
50	74.14	57.644±2.807	18.411±0.877	39.232±3.139	0.148±0.003
50	60	53.602±1.055	12.124±0.010	41.478±1.066	0.150±0.000
50	60	55.986±0.621	18.616±0.755	37.369±1.220	0.107±0.012
50	60	54.663±0.624	10.483±0.841	44.179±1.445	0.137±0.003

Table 3: Statistical analysis of the effects of temperature and sucrose concentration on the responses WL, SG, ML and SGR in the osmotic dehydration of jabuticaba peel.

Response	Factors	Effect	SD	t(27)	P
WL	Average	54.799	0.922	59.428	0.000
	Temperature (°C) (L)	0.988	1.129	0.874	0.390
	Temperature (°C) (Q)	4.121	1.345	3.065	0.005
	Sucrose concentration (°Brix) (L)	8.782	1.129	7.776	0.000
	Sucrose concentration (°Brix) (Q)	-3.885	1.345	-2.890	0.008
	Temperature (°C) x sucrose concentration (°Brix) (L)	-0.991	1.597	-0.620	0.540
SG	Average	10.712	0.843	12.700	0.000
	Temperature (°C) (L)	-0.476	1.033	-0.460	0.649
	Temperature (°C) (Q)	1.485	1.230	1.208	0.238
	Sucrose concentration (°Brix) (L)	5.953	1.033	5.763	0.000
	Sucrose concentration (°Brix) (Q)	0.914	1.230	0.743	0.464
	Temperature (°C) x sucrose concentration (°Brix) (L)	0.176	1.461	0.120	0.905
ML	Average	44.088	1.398	31.530	0.000
	Temperature (°C) (L)	1.463	1.713	0.854	0.400
	Temperature (°C) (Q)	2.636	2.039	1.293	0.207
	Sucrose concentration (°Brix) (L)	2.829	1.713	1.652	0.110
	Sucrose concentration (°Brix) (Q)	-4.799	2.039	-2.354	0.026

	Temperature (°C) x sucrose concentration (°Brix) (L)	-1.166	2.421	-0.482	0.634
	Average	0.131	0.0039	33.562	0.000
	Temperature (°C) (L)	0.021	0.0047	4.349	0.000
	Temperature (°C) (Q)	-0.011	0.0057	-1.878	0.071
SGR	Sucrose concentration (°Brix) (L)	0.040	0.0048	8.325	0.000
	Sucrose concentration (°Brix) (Q)	-0.006	0.0057	-0.999	0.326
	Temperature (°C) x sucrose concentration (°Brix) (L)	0.013	0.0068	1.970	0.059

L: linear; Q: quadratic; SD: standard deviation; 95% of significance ($p \leq 0.05$).

Table.4: Analysis of variance of the polynomial adjusted for WL, SG, ML and SGR responses in osmotic dehydration of jabuticaba.

Response	Source of Variation	SS	DF	MS	F _{calc}	F _{tab}	R ²
WL	Regression	654.9757	3	218.3252	29.3914	2.9340	0.7525
	Residual	215.4180	29	7.4282	-	-	
	Lack-of-fit	130.7292	5	26.1458	7.4195	2.6207	
	Pure error	84.6888	24	3.5287	-	-	
	Total	870.3937	32	-	-	-	
SG	Regression	212.6216	1	212.6216	35.6825	4.1620	0.5351
	Residual	184.7220	31	5.9587	-	-	
	Lack-of-fit	155.9327	7	22.2761	18.5696	2.4226	
	Pure error	28.7896	24	1.1996	-	-	
	Total	397.3436	32	-	-	-	
ML	Regression	144.0063	1	144.0063	7.8395	4.162	0.2018
	Residual	569.4435	31	18.3691	-	-	
	Lack-of-fit	415.7771	7	59.3967	9.2767	2.4226	
	Pure error	153.6664	24	6.4028	-	-	
	Total	713.4498	32	-	-	-	
SGR	Regression	0.0121	2	0.0060	30.0000	3.3158	0.7181
	Residual	0.0048	30	0.0002	-	-	
	Lack-of-fit	0.0017	6	0.0003	3.0000	2.5082	
	Pure error	0.0031	24	0.0001	-	-	
	Total	0.0169	32	-	-	-	

SS: sum of squares; DF: degree of freedom; MS: mean square; F_{tab}: tabulated values of F at $p \leq 0.05$.

Table.5: Coefficients of the mathematical model for WL, SG, ML and SGR responses in osmotic dehydration of jabuticaba peel.

Y	β_1	β_2	β_3	β_4	β_5	β_6
WL (%)	-7.3060	-1.7138	0.0206	3.0181	-0.0194	-0.0049
SG (%)	31.6942	-0.8189	0.0074	-0.2948	0.0046	0.0009
ML (%)	-39.0002	-0.8948	0.0132	3.3129	-0.0240	-0.0058
SGR (°Brix h ⁻¹)	-0.0766	0.0024	-0.0001	0.0021	-0.0001	0.0001

Table.6: Levels of proximate composition, total, reducing and non-reducing sugars, total soluble solids, ph, titratable acidity, water activity, phenolic compounds and antioxidant potential of dried jaboticaba peel.

Analysis	Mean \pm SD
Moisture (g 100 g ⁻¹)	24,902 \pm 0,461
Ash (g 100 g ⁻¹)	0,231 \pm 0,019
Lipids (g 100 g ⁻¹)	0,306 \pm 0,046
Proteins (g 100 g ⁻¹)	2,100 \pm 0,006
Carbohydrates (g 100 g ⁻¹)	72,461 \pm 0,486
Total sugars* (g 100 g ⁻¹)	66.945 \pm 0.193
Reducing sugars* (g 100 g ⁻¹)	58.300 \pm 0.911
Non-reducing sugars (g 100 g ⁻¹)	8.645 \pm 0.722
Total soluble solids (°Brix)	67.667 \pm 0.547
Total titratable acidity (g 100 g ⁻¹)	9.669 \pm 0.354
pH	3.407 \pm 0.006
Water activity	0.665 \pm 0.007
Phenolic compounds (mg GAE 100 g ⁻¹)	
Ether extract	2.258 \pm 0.372
Ethanolic extract	73.448 \pm 9.400
Aqueous extract	348.315 \pm 2.864
Antioxidant potential(% DPPH discoloration)	
Ether extract	16.667 \pm 1.91
Ethanolic extract	21.273 \pm 0.214
Aqueous extract	24.770 \pm 0.657

*Expressed as glucose.

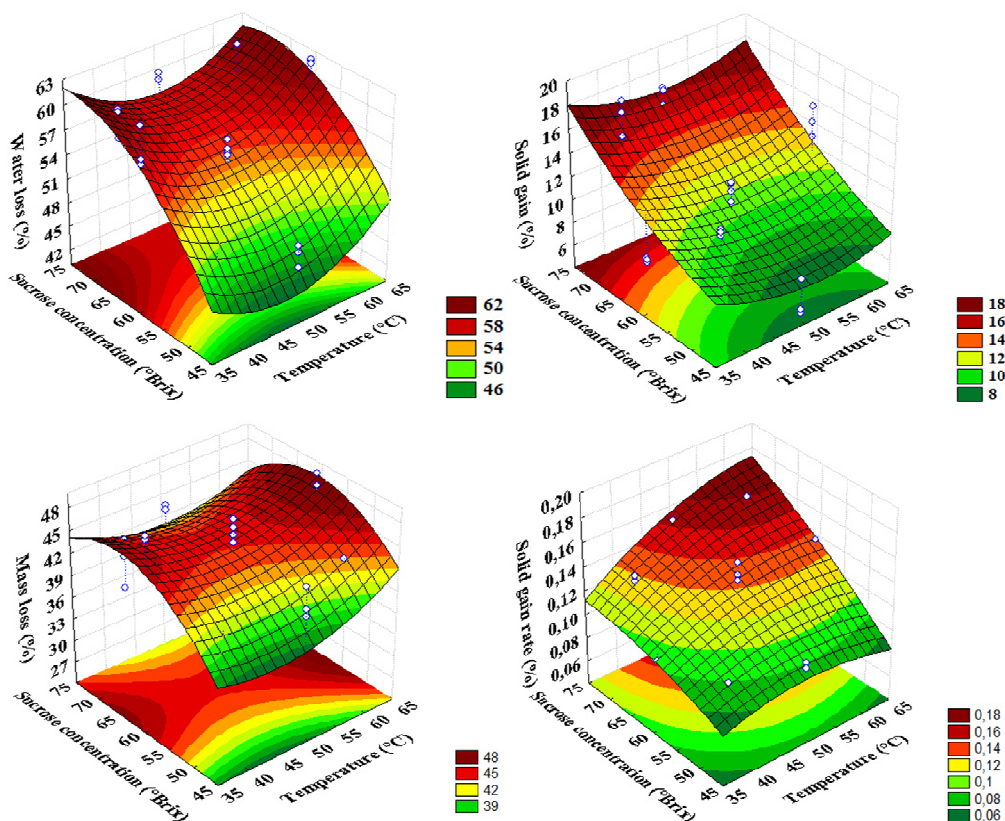


Fig.1: Response surface of WL, SG, ML and SGR on osmotic dehydration of jaboticaba peel as a function of temperature and sucrose concentration.